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**Attachments:** 2014-05-23 FMC transmittal GW Bench Treatability Study Work Plan.pdf; 2014-05-23 FMC Bench-Scale Treatability Study Work Plan - Groundwater RD....pdf

Please see attached.

# **Bench-Scale Treatability Study Work Plan**

## **Groundwater Remedial Design for the FMC OU**

**May 23, 2014**

### **1.0 INTRODUCTION/PURPOSE**

In accordance with the Unilateral Administrative Order (UAO) for Remedial Design and Remedial and Remedial Action at the FMC OU and as described in the Remedial Design Work Plan, FMC is proceeding with bench-scale treatability testing in association with the remedial design (RD) for the groundwater remedial action for the FMC OU. This work plan describes the bench-scale treatability testing and laboratory analytical procedures to be followed to evaluate the effectiveness of various treatment process steps and provide guidance on design parameters for a groundwater treatment facility pursuant to the selected groundwater remedy treatment option B for the FMC OU.

MWH has retained FLSmidth (FLS) as a subcontractor to perform bench scale treatability testing to evaluate and optimize a coagulation/flocculation process and to evaluate an alternative lime softening process. FMC has also retained Parsons Treatability Lab (Parsons) as an independent testing firm. The coagulation/flocculation jar testing described in this work plan will also be performed by Parsons. ALS Laboratory, a NELAP accredited laboratory, will perform the laboratory analyses of all pre- and post-treatability test samples. The bench-scale treatability testing will be performed using the bulk groundwater samples collected during the 72-hour aquifer (pump) testing pursuant to the Extraction Zone Hydrogeologic Study Work Plan and thus this testing program will not involve any field work on site at the FMC OU.

The primary objective of the treatability testing is to determine the most appropriate coagulant type, coagulant dose, required mixing rates, and optimum pH to lower the arsenic level below the current EPA drinking water standard of 10 µg/L. The focus of the treatability study is arsenic due to the fact that arsenic is the primary risk-driving constituent in groundwater at the FMC OU and, as shown on Table 1, is the only constituent in the extracted groundwater that is expected to exceed its comparative value (CV). The treatability study will also evaluate removal of total phosphorus which, although there is no CV, will be reduced to a target level of 0.3 mg/L as described in the SFS Report.

The sludge produced during the treatability testing will be analyzed for RCRA-8 Metals (arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver) using the toxicity characteristic leaching procedure (TCLP) to determine whether the sludge will likely be characterized as hazardous or non-hazardous.

In summary, the tasks to be performed in support of the treatability study consist of the following:

- Analysis of raw groundwater to determine the characteristics of the water, with particular emphasis on measuring the concentrations of arsenic in its various valence states (i.e. As(III) and As(V))

**TABLE 1**  
**Estimated Average Extracted Groundwater Quality Vs Comparative Values**

PARAMETER	WELL-110	WELL-146	TW-9S	Average Concentration of Constituent	Comparative Value <sup>1</sup>
Sampling Event Date	4thQ2006 thru 2ndQ2008	4thQ2006 thru 2ndQ2008	4thQ2006 thru 2ndQ2008		
<b><u>Field Measurements</u></b>					
pH (Field)	6.86	7.11	7.06	<b>7.01</b>	<b>6.5-8.5</b>
SC (UMHOS/CM)	1429	1294	1842	<b>1521.7</b>	<b>NA</b>
<b><u>General WQP (mg/L)</u></b>					
Potassium	25.4	46.3	58.4	<b>43.4</b>	<b>NA</b>
Sulfate	215	128	161	<b>168.0</b>	<b>250</b>
Chloride	89.8	132	187	<b>136.3</b>	<b>250</b>
Fluoride	0.44	0.38	0.10	<b>0.30</b>	<b>4.0</b>
Ammonia	0.15	0.15	0.2	<b>0.17</b>	<b>NA</b>
Nitrate	3.8	6.13	9.95	<b>6.63</b>	<b>10</b>
Orthophosphate/ Total Phosphorus	3.09	1.33	3.21	<b>2.54</b>	<b>NA</b>
<b><u>Metals (mg/L)</u></b>					
Arsenic	<b>0.048</b>	<b>0.029</b>	<b>0.027</b>	<b>0.03</b>	<b>0.01</b>
Cadmium	<0.0005	<0.0005	<0.0005	<b>&lt;0.0005</b>	<b>0.005</b>
Copper <sup>2</sup>	0.0015	<0.025	0.0011	<b>0.001</b>	<b>1</b>
Cyanide <sup>3</sup>	<0.01	0.011	<0.01	<b>0.01</b>	<b>0.2</b>
Lead <sup>2</sup>	<0.003	<0.003	<0.003	<b>&lt;0.003</b>	<b>TT, 0.015</b>
Mercury <sup>2</sup>	<0.0002	<0.0002	<0.0002	<b>&lt;0.0002</b>	<b>0.0006</b>
Nickel <sup>2</sup>	<0.04	<0.04	<0.04	<b>&lt;0.04</b>	<b>0.73</b>
Silver <sup>2</sup>	<0.005	<0.005	<0.005	<b>&lt;0.005</b>	<b>0.1</b>
Selenium	0.029	0.003	0.005	<b>0.012</b>	<b>0.05</b>
Zinc <sup>2</sup>	0.00036	0.0024	0.00037	<b>0.001</b>	<b>5</b>

<sup>1</sup> - The Comparative Values (CVs) are taken from Table 4.2-1 "Groundwater Representative Concentrations and Comparative Values" in the GWCCR, June 2009 Final. Comparative values are primary MCLs, treatment technology standards or secondary standards.

<sup>2</sup> - Results from November 2001 Special Groundwater sampling event.

<sup>3</sup> - Results from May 2008 Special Groundwater sampling event.

Yellow highlight indicates groundwater concentration exceeds CV.

- Bench-scale jar testing to determine the most appropriate coagulant, dosing rate, mixing rate, and pH required for effective removal of arsenic to below the current EPA drinking water standard of 10 µg/L, with a secondary goal to reduce total phosphorous levels
- Water quality characterization of the supernatant to measure treatment effectiveness
- Testing to determine the physical and chemical characteristics of the precipitate (e.g. sludge)

This work plan is primarily focused on testing procedures to optimize a coagulation/flocculation treatment process. FLS has proposed conducting additional bench-scale testing to evaluate the effectiveness of a lime softening process as an alternative to a coagulation/flocculation process. The methods for bench-scale testing of the lime softening process will be conducted according to FLS’s standard procedures, and as such are not specified in this work plan.

### 2.0 GROUNDWATER SAMPLES

MWH will provide FLS and Parsons each with a minimum of 13 gallons (50 liters) of the bulk groundwater samples collected during the 72-hour aquifer (pump) testing pursuant to the Extraction Zone Hydrogeologic Study Work Plan (EZHP). As specified in the EZHP, a sample from the 72-hour composite bulk samples was submitted to ALS laboratory for analyses per the EZHP quality assurance project plan (QAPP) and Table 3-2 of the EZHP. Those validated analytical results will be utilized in this study as representing the raw (untreated) water quality.

### 3.0 JAR TESTING

Jar testing methods will generally conform to ASTM D2035-13 (Attachment A), except where stated differently in this work plan. Jar testing will be performed in batches of 5 jars, with each individual jar using 1 L of source water. Multiple batches of jar tests will be conducted to incrementally optimize dose, mixing scheme, and pH, as shown in Table 2.

**TABLE 2**  
**Schedule of Jar Testing**

Step	Coagulant Dose	Mixing	pH
1	Variable (Table 2)	M <sub>1</sub> (Table 3)	Not adjusted
2	Optimum from Step 1	Variable (Table 3)	Not adjusted
3	Optimum from Step 1	Optimum from Step 2	6, 7, 7.5, 8, and 9

Jar testing will be conducted using the following chemical coagulants:

- Ferric Chloride (FeCl<sub>3</sub>)
- Aluminum sulfate (Alum; Al<sub>2</sub>(SO<sub>4</sub>)<sub>3</sub>)

A concentrated stock solution of each coagulant type should be freshly made or obtained prior to conducting jar testing. The volume of stock solution required to achieve the desired coagulant dose for a 1 L total sample volume should be calculated for each coagulant dose.

All testing should be conducted with the source water at  $60 \pm 5$  degrees F, the expected temperature that the groundwater will enter the full scale treatment process. Water temperature should be measured and recorded (from an individual jar, not from the bulk sample) for each set of jar tests conducted.

Visual observations should be recorded throughout each batch of jar testing, including observations regarding the timing of floc formation, size of floc, and settling rate.

### 3.1 Step 1: Dose Optimization (minimum of two batches of 5 jars = 10 L)

Using the mixing scheme  $M_1$  shown in Table 3 and without adjusting the pH of the source water, test 5 separate jars (for each coagulant) with each coagulant dose presented in Table 3. If sufficient flocculation is not observed at the highest proposed dose of 40 mg/L, an additional batch of jars should be tested with higher doses (i.e., 50 mg/L, 60 mg/L, etc.) until sufficient floc formation is observed.

**TABLE 3**  
**Proposed Coagulant Doses**

Coagulant Dose (mg/L as $\text{FeCl}_3$ or mg/L as $\text{Al}_2(\text{SO}_4)_3$ )
5
10
20
30
40

Once the slow mixing has ended, a turbidity reading should be collected every 5 minutes from each jar in order to measure the floc settling rate. Turbidity samples should be collected using a pipette or sampling port to draw a supernatant sample from roughly half of the total sample depth, and should be measured immediately using a turbidimeter. These measurements should be taken until 20 minutes after commencement of the slow mix period (i.e., a total of 4 turbidity measurements per jar). At the discretion of the engineer or technician performing the testing, additional turbidity measurements (beyond 20 minutes) may be beneficial.

At 20 minutes after the commencement of slow mixing, a 30 mL supernatant sample should be collected from each jar for supernatant water quality analysis, as presented in Section 4. The pH of this sample should also be measured and recorded.

Once the Step 1 jar testing is complete and analytical results for the supernatant samples are reviewed, an optimum coagulant dose will be selected based on measured As removals. The optimum dose will be selected as the minimum dose where an increase in dose does not result in a significant increase in As removal.

### 3.2 Step 2: Mixing Optimization (2 batches of 4 jars = 8 L)

Each batch of Step 2 testing will use 4 jars; two jars will be dosed with  $\text{FeCl}_3$  and two will be dosed with  $\text{Al}_2(\text{SO}_4)_3$ . The optimum dose (determined in Step 1) and the next lowest dose will be used for each coagulant type. Two doses are used for each coagulant type in order to better compare the effect of various mixing schemes on floc formation and setting rate.

Without adjusting the source water pH, test the two remaining mixing schemes presented in Table 4 (i.e.,  $M_2$  and  $M_3$ ). The purpose of the step is to compare the effectiveness of various mixing schemes.

**TABLE 4**  
**Mixing Schemes**

Mixing Scheme	Rapid Mix		Slow Mix 1		Slow Mix 2	
	Minutes	<i>rpm</i>	Minutes	<i>rpm</i>	Minutes	<i>rpm</i>
$M_1$	1	100	30	45	-	-
$M_2$	1	100	15	45	15	20
$M_3$	0.5	100	15	45	15	20

Again, collect turbidity readings from each jar every 5 minutes over a 20 minute setting period to measure the setting rate. Collect a 30 mL supernatant sample from each jar at the end of the 20 minute setting period, measure the pH of each sample, and submit the sample for As and Total phosphorous water quality analysis.

Out of the three mixing schemes tested, the optimum mixing scheme will be selected for each coagulant type based on the measured turbidity and As removal after the 20 minute setting period as well as the overall turbidity profile (i.e., setting rate).

### 3.3 Step 3: pH Optimization (2 batches of 5 jars = 10 L)

Using the optimum dose selected in Step 1 and the optimum mixing scheme selected in Step 2, test the effect of variable pH on coagulant/flocculation. Use NaOH to increase pH and HCl to decrease pH of the source water to 5 separate pH target points of: 6, 7, 7.5, 8, and 9. Each pH should be within  $\pm 0.10$  pH units of the target prior to beginning the jar testing.

Again, collect turbidity readings from each jar every 5 minutes over a 20 minute setting period to measure the setting rate. Collect a 30 mL supernatant sample from each jar at the end of the 20 minute setting period, measure the pH of each sample, and submit the sample for As and total phosphorous water quality analysis.

### 3.4 Step 4: Optimum Conditions (2 batches of 5 jars = 10 L)

In order to determine the reproducibility of treatment at the optimum conditions determined by Steps 1 through 3, and also to generate a sufficient volume of sludge for solids analysis, 5 jars will be run using the optimum dose, mixing, and pH for each of the coagulants. If it is clear that one coagulant greatly outperforms the other, this step may only be conducted for one of the coagulants.

At the end of the 20 minute setting period, a 30 mL sample should be collected from each jar, and a 2 L composite sample should be collected using 400 mL from each of the 5 jars.

The jars should then be left undisturbed beyond the 20 minute setting period, until no additional setting is observed. The remaining supernatant should then be decanted from each jar, without disturbing the sludge blanket at the bottom of the jar.

The mass of solids generated per volume of treated water should also be measured and recorded for the each type of coagulant. This may be achieved by drying (at 105 degrees C) and weighing a measured portion (i.e., percent volume) of the resulting sludge (preferably prior to filterability testing).

Filterability testing should be conducted on the decanted sludge (sludge from each of the 5 jars can be combined into 1 bulk sludge sample). Filter testing methods are left to the discretion of FLS and Parsons, depending on the bench scale filter testing equipment available. This testing may include an analysis of particle size distribution and/or performance of leaf-filter testing.

The remaining filter cake from the treatability testing should be collected and submitted for laboratory analysis, as described in Section 4.

## **4.0 LABORATORY ANALYSIS**

The liquid and solid samples collected during the treatability study will be analyzed at ALS, a NELAP-accredited analytical laboratory. Sample collection, submission, and analysis will be conducted according to the attached Quality Assurance Project Plan (Attachment B) and as summarized below.

### **4.1 Water Quality Analysis**

All 30 mL treated water (supernatant) samples collected as part of the Step 1 through 4 testing (anticipated total of 38 samples) will be submitted for laboratory analysis of As and total phosphorous. The 30 mL supernatant samples will be submitted for analysis immediately after each step of jar testing is complete, and a 2-day turnaround time will be requested, so that analytical results can be reviewed prior to moving on to the next step of jar testing.

Each of the 2 L composite supernatant samples (maximum of 2) collected as part of the Step 4 treatment testing will be submitted for a more extensive list of water quality parameters, as described in Attachment B.

### **4.2 Solids Analysis**

For the Step 4 testing of each coagulant type, the filter cake remaining from the treatability testing should be collected and submitted for laboratory analysis of RCRA-8 Metals (arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver) using the toxicity characteristic leaching procedure (TCLP) to determine whether the sludge is characterized as hazardous or non-hazardous

and whether it will require special disposal. A minimum sample size of 100 g is required for this procedure.

The mass of solids generated per volume of treated water should also be measured and recorded for the each type of treatment (i.e., including softening) and each type of coagulant, under optimum conditions.

## **5.0 DATA AND REPORTING**

All data collected during this bench-top treatability study for the groundwater remedy will be retained by FMC and/or its contractors consistent with the records retention requirements under the UAO. The data and analytical laboratory results collected in direct support of this bench-top treatability study will be reported to EPA in the Groundwater Remedy Preliminary (30%) Design Submittal that is scheduled for submittal in December 2014 per the Groundwater RD/RA Preliminary Schedule (Rev. 5.2) submitted with the April 2014 UAO monthly report.

**ATTACHMENT A**

**ASTM D2035-13: STANDARD PRACTICE FOR COAGULATION-FLOCCULATION  
JAR TEST OF WATER**



## Standard Practice for Coagulation-Flocculation Jar Test of Water<sup>1</sup>

This standard is issued under the fixed designation D2035; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon ( $\epsilon$ ) indicates an editorial change since the last revision or reapproval.

### 1. Scope

1.1 This practice covers a general procedure for the evaluation of a treatment to reduce dissolved, suspended, colloidal, and nonsettleable matter from water by chemical coagulation-flocculation, followed by gravity settling. The procedure may be used to evaluate color, turbidity, and hardness reduction.

1.2 The practice provides a systematic evaluation of the variables normally encountered in the coagulation-flocculation process.

1.3 The values stated in SI units are to be regarded as standard.

1.4 *This standard does not purport to address the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

### 2. Referenced Documents

2.1 *ASTM Standards:*<sup>2</sup>

D1129 Terminology Relating to Water

D1193 Specification for Reagent Water

D1293 Test Methods for pH of Water

D3370 Practices for Sampling Water from Closed Conduits

D6855 Test Method for Determination of Turbidity Below 5 NTU in Static Mode

D7315 Test Method for Determination of Turbidity Above 1 Turbidity Unit (TU) in Static Mode

### 3. Terminology

3.1 *Definitions*—For definitions of terms used in this practice, refer to Terminology D1129.

<sup>1</sup> This practice is under the jurisdiction of ASTM Committee D19 on Water and is the direct responsibility of Subcommittee D19.03 on Sampling Water and Water-Formed Deposits, Analysis of Water for Power Generation and Process Use, On-Line Water Analysis, and Surveillance of Water.

Current edition approved Jan. 1, 2013. Published February 2013. Originally approved in 1964. Last previous edition approved in 2008 as D2035–08. DOI: 10.1520/D2035-13.

<sup>2</sup> For referenced ASTM standards, visit the ASTM website, [www.astm.org](http://www.astm.org), or contact ASTM Customer Service at [service@astm.org](mailto:service@astm.org). For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

### 4. Summary of Practice

4.1 The coagulation-flocculation test is carried out to determine the chemicals, dosages, and conditions required to achieve optimum results. The primary variables to be investigated using the recommended practice include, but are not limited to:

4.1.1 Chemical additives,

4.1.2 pH,

4.1.3 Temperature, and

4.1.4 Order of addition and mixing conditions.

### 5. Significance and Use

5.1 This practice permits the evaluation of various coagulants and coagulant aids used in the treatment of water and waste water for the same water and the same experimental conditions.

5.2 The effects of concentration of the coagulants and coagulant aids and their order of addition can also be evaluated by this practice.

### 6. Interferences

6.1 There are some possible interferences that may make the determination of optimum jar test conditions difficult. These include the following:

6.1.1 *Temperature Change (During Test)*—Thermal or convection currents may occur, interfering with the settling of coagulated particles. This can be prevented by temperature control.

6.1.2 *Gas Release (During Test)*—Flotation of coagulated floc may occur due to gas bubble formation caused by mechanical agitator, temperature increase or chemical reaction.

6.1.3 *Testing-Period*—Biological activity or other factors may alter the coagulation characteristics of water upon prolonged standing. For this reason the period between sampling and testing should be kept to a minimum, with the time being recorded.

### 7. Apparatus

7.1 *Multiple Stirrer*—A multiposition stirrer with continuous speed variation from about 20 to 150 rpm should be used. The stirring paddles should be of light gage corrosion-resistant material all of the same configuration and size. An illuminated base is useful to observe the floc formation. Precautionary

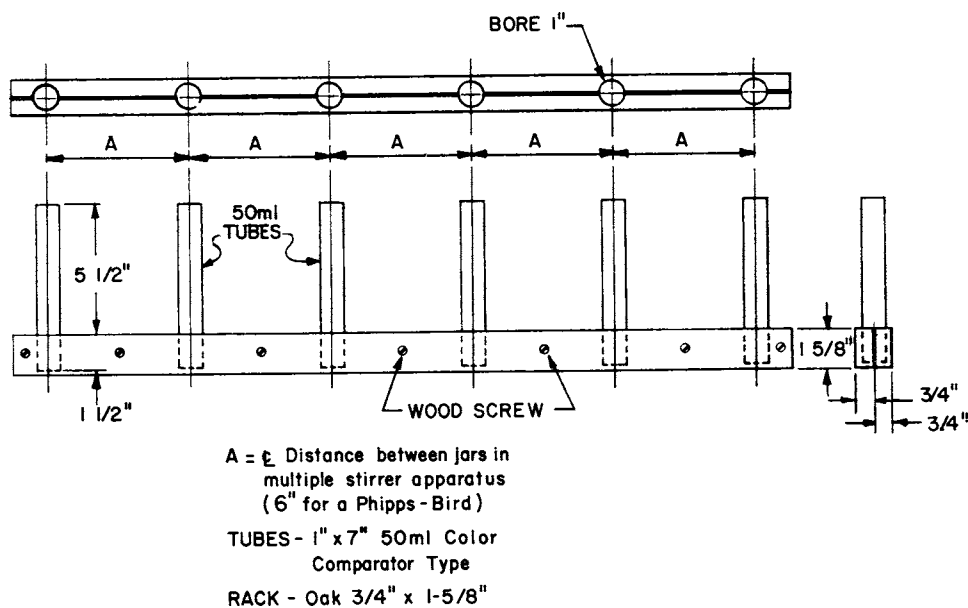


FIG. 1 Reagent Rack for Multiple Stirrer Jar Test Apparatus

measures should be taken to avoid heat being imparted by the illumination system which may counteract normal settling.

7.2 *Jars (or Beakers)*, all of the same size and shape; 1500-mL Griffin beakers may be used (1000-mL recommended minimum size).

7.3 *Reagent Racks*— A means of introducing each test solution to all jars simultaneously. There should be at least one rack for each test solution or suspension. The racks should be similar to that shown in Fig. 1.

## 8. Reagents

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.<sup>3</sup> Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Reference to water that is used for reagent preparation, rinsing or dilution shall be understood to mean water that conforms to the quantitative specifications of Type IV reagent water of Specification D1193.

8.3 The following chemicals and additives are typical of those used for test solutions and suspensions. The latter, with the exception of coagulant aids, may be prepared daily by mixing chemicals with water to a concentration of 10 ( $\pm 0.1$ ) g/L (1.0 mL of test solution or suspension when added to 1 L of sample is equivalent to 10 mg/L):

- Prime Coagulants
  - Alum [ $\text{Al}_2(\text{SO}_4)_3 \cdot 18\text{H}_2\text{O}$ ]
  - Ferric sulfate [ $\text{Fe}_2(\text{SO}_4)_3 \cdot x\text{H}_2\text{O}$ ]
  - Ferric chloride ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ )
  - Ferrous sulfate ( $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ )
  - Magnesium carbonate ( $\text{MgCO}_3 \cdot 3\text{H}_2\text{O}$ )
  - Sodium aluminate ( $\text{NaAlO}_2$ )
- Coagulant Aids
  - Activated silica
  - Anionic (polyelectrolyte)
  - Cationic (polyelectrolyte)
  - Nonionic Polymer
- Oxidizing Agents
  - Chlorine ( $\text{Cl}_2$ )
  - Chlorine dioxide ( $\text{ClO}_2$ )
  - Potassium permanganate ( $\text{KMnO}_4$ )
  - Calcium hypochlorite [ $\text{CaCl}(\text{ClO}) \cdot 4\text{H}_2\text{O}$ ]
  - Sodium hypochlorite ( $\text{NaClO}$ )
- Alkalis
  - Calcium carbonate ( $\text{CaCO}_3$ )
  - Dolomitic lime
  - (58 %  $\text{CaO}$ , 40 %  $\text{MgO}$ )
  - Lime, hydrated [ $\text{Ca}(\text{OH})_2$ ]
  - Magnesium oxide ( $\text{MgO}$ )
  - Sodium carbonate ( $\text{Na}_2\text{CO}_3$ )
  - Sodium hydroxide ( $\text{NaOH}$ )
- Weighting Agents
  - Bentonite
  - Kaolin
  - Other clays and minerals
- Miscellaneous
  - Activated carbon (powdered)

8.4 *Coagulant Aids*— There are numerous commercially available coagulant aids or polyelectrolytes. All polyelectrolytes are classified anionic, cationic or nonionic, depending upon their composition. These aids may have the ability to produce large, tough, easily-settled floc when used alone or in conjunction with inorganic coagulants. A small dosage (under 1 mg/L) may permit a reduction in the dosage of, or complete elimination of, the coagulant. In the latter case, the polyelectrolyte would be considered the prime coagulant rather than a coagulant aid. Aids come in powdered and liquid form. Powdered aids should be prepared as 0.1 % solutions with appropriate aliquots to provide proper dosage. Always add

<sup>3</sup> *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD.

powdered aids to the dissolving water rather than the reverse, and add slowly to the shoulder of a vortex created by stirring. If a vortex is not formed, the dry powder will merely collect on the surface of the water in gummy masses and become very difficult to dissolve. Dissolving time may vary from several minutes to several hours. Suggested manufacturers' procedures for wetting, dissolving, and storing should be followed when available. Liquid forms can be readily prepared to the above strength without difficulty.<sup>4</sup>

## 9. Sampling

9.1 Collect the water sample under test in accordance with the applicable Practices **D3370**.

## 10. Procedure

10.1 Measure equal volumes (1000 mL) of sample into each of the jars or 1500-mL Griffin beakers. As many sample portions may be used as there are positions on the multiple stirrer. Locate beakers so that the paddles are off-center, but clear the beaker wall by about 6.4 mm ( $\frac{1}{4}$  in.). Record the sample temperature at the start of the test.

10.2 Load the test chemicals in the reagent racks. Use one rack for each series of chemical additions. Make up each tube in the rack to a final volume of 10 mL, with water, before using. There may be a situation where a larger volume of reagent will be required. Should this condition prevail, fill all tubes with water to a volume equal to the largest volume of reagent in the reagent rack. When adding slurries, it may be necessary to shake the rack to produce a swirling motion just prior to transfer.

10.3 Start the multiple stirrer operating at the "flash mix" speed of approximately 120 rpm. Add the test solution or suspensions, at predetermined dosage levels and sequence. Flash mix for approximately 1 min after the additions of chemicals. Record the flash mix time and speed (rpm).

10.4 Reduce the speed as necessary to the minimum required to keep floc particles uniformly suspended throughout

the "slow mix" period. Slow mix for 20 min. Record the time for the first visible floc formation. Every 5 min (during the slow mix period), record relative floc size and mixer speed (rpm). If coagulant aids are used, mixing speed is critical because excessive stirring tends to break up early floc formation and may redisperse the aid.

10.5 After the slow mix period, withdraw the paddles and observe settling of floc particles. Record the time required for the bulk of the particles to settle. In most cases this time will be that required for the particles to settle to the bottom of the beaker; however, in some cases there may be interfering convection currents. If so, the recorded settling time should be that at which the unsettled or residual particles appear to be moving equally upward and downward.

10.6 After 15 min of settling, record the appearance of floc on the beaker bottom. Record the sample temperature. By means of a pipet or siphon, withdraw an adequate sample volume of supernatant liquor from the jar at a point one half of the depth of the sample, to conduct color,<sup>5</sup> turbidity, pH and other required analyses, (**Note 1**) determined in accordance with Test Methods **D6855** or **D7315** (for turbidity) and **D1293** (for pH). A suggested form for recording results is appended (see **Fig. 2**).

**NOTE 1**—Tests for residual chemicals should be included, for example, alum; residual  $\text{Al}_2\text{O}_3$ ; copperas; residual  $\text{Fe}_2\text{O}_3$ ; etc.

10.7 Repeat steps **10.1-10.6** until all pertinent variables have been evaluated.

10.8 The times given in **10.3**, **10.4**, and **10.6** are only suggestions.

## 11. Reproducibility

11.1 It is recognized that reproducibility of results is important. To demonstrate reproducibility, the so-called 3 and 3 procedure is suggested. In this procedure, duplicate sets of 3 jars each are treated simultaneously with the same chemical dosages in jars 1 and 4, 2 and 5, and 3 and 6.

## 12. Keywords

12.1 coagulation; flocculation; jar tests

<sup>4</sup> A periodically updated "Report on Coagulant Aids for Water Treatment" is published by the Environmental Protection Agency Office of Water Supply, Cincinnati, Ohio 45268, listing coagulant aids that may be used in water treatment without adverse physiological effects on those using the water, based on information submitted by the manufacturers or distributors, or both.

<sup>5</sup> For the color determination, reference is made to *Standard Methods for the Examination of Water and Waste Water*, Fourteenth edition, American Public Health Association, Inc., New York, NY, 1975, pp. 64–71.

[illegible]

### FIG. 2 Jar Test Data

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**ATTACHMENT B**

**QUALITY ASSURANCE PROJECT PLAN (QAPP)**

# QUALITY ASSURANCE PROJECT PLAN

## B.1 INTRODUCTION

This attachment presents the Quality Assurance Project Plan (QAPP) for the groundwater bench-scale treatability study samples for analytical laboratory analyses.

## B.2 DATA QUALITY OBJECTIVES

During execution of the Treatability Study, two types of data to be collected:

1. Data on reagent types and addition rates utilized during the bench-top testing; and
2. Analyses of liquid samples collected prior to and after bench-top testing and analysis of solid (filter cake) samples collected after the bench-top testing.

### B.2.1 Bench Notes

If chemical coagulant stock solutions are made in-house, the amounts and chemical formulas of all reagents used to create stock solutions should be recorded. The volume of stock solution added to each jar to obtain the desired doses should also be recorded.

Visual observations should be recorded throughout each batch of jar testing, including observations regarding the timing of floc formation, size of floc, and settling rate.

For each jar, turbidity readings should be measured and recorded every 5 minutes over the 20 minuet setting period. The pH for the samples collected at the end of the 20 minuet setting period should be measured and recorded.

For pH optimization testing, the volume of NaOH and HCl added to each 1 L sample to reach the pH target points should be recorded.

Detailed documentation of volumes, drying temperature and times, and weights collected to measure the mass of solids generated per volume of treated water should be recorded.

Detailed documentation should also be recorded describing the methods and results for any other testing conducted, including softening testing and filterability testing.

### B.2.2 Treatability Samples - Laboratory Analyses

Samples will be collected for laboratory analyses. The liquid and solid samples collected during the treatability study will be analyzed at a NELAP-accredited analytical laboratory. The liquid samples will be analyzed for the all or some of the parameters listed in Table B-2 and the solid samples will be analyzed for the TCLP metals as listed in Table B-3. Samples will be collected and handled as described in Section B.3 below. The specified reporting limits are below the lower of the groundwater cleanup standard or comparative values (for parameters with no cleanup standard) to assure the data are useable.

## B.3 SAMPLING/MEASUREMENT PROCEDURES

### B.3.1 Liquid and Solid Sampling Procedures

As described in Section 3 of the Work Plan, liquid (supernatant) and solid (settled sludge) samples will be collected during the treatability study. The following samples are anticipated:

- Raw (untreated) water
- Treated water samples (supernatant)
- Settled Sludge (filtered from decanted jars)

Two raw water samples will be collected and analyzed for As(III) and As (V). All 30 mL treated water (supernatant) samples collected as part of the Step 1 through 4 testing (anticipated total of 38 samples) will be submitted for laboratory analysis of As and total phosphorous. The 30 mL supernatant samples will be submitted for analysis immediately after each step of jar testing is complete, and a 2-day turnaround time will be requested, so that analytical results can be reviewed prior to moving on to the next step of jar testing.

Each of the 2 L composite supernatant samples (maximum of 2) collected as part of the Step 4 (Optimized) treatment testing will be submitted for the full list of parameters presented in Table B-2 except As(III) and As(V) analysis will not be performed.

Solid samples of the settled sludge will be collected in 250 mL jars and submitted for analysis for the RCRA metals using the TCLP method as shown on Table B-3.

The procedures for collecting, labeling and handling these samples is described below.

### B.3.2 Sample Designation

All collected samples will be labeled in a clear and precise way for proper identification and tracking in the laboratory. Each sample will be labeled and recorded in the logbook as follows:

FMC TS

[Testing Company]

[Date, Time]

[Treatment Testing Step #, Coagulant Type, Coagulant Dose (mg/L), Mixing Scheme, pH (if adjusted)]

Example:

FMC TS

FLS

6/16/2014, 13:05

Step 1, Alum, 30, M<sub>1</sub>

### B.3.3 Sample Collection

For each step of jar testing, a 30 mL sample should be collected from each jar at the end of the 20 minute setting period. A 2 L composite sample should be collected from each batch of Step 4 testing by collecting 400 mL from each of the 5 jars. All supernatant samples should be collected using a pipette or sampling port to draw a sample from roughly half of the depth of liquid in the jar. Care should be taken to ensure that the settled sludge blanket is not disturbed and drawn into the supernatant sample.

A minimum sample size of 100 g of filtered sludge should be submitted for TCLP analysis (Table B-3) for each batch of Step 4 testing (maximum of 2; one for ferric chloride and one for alum).

The recommended sample containers and required sample preservation and holding times for the liquid samples to be submitted for laboratory analysis are summarized in the inset table below.

**TABLE B-1**  
**Sample Preservation and Holding Time Requirements for Laboratory Analyses**

Parameter	Recommended Container	Preservative	Maximum Holding Time
Water Quality (Cl <sup>-</sup> , F <sup>-</sup> , NO <sub>3</sub> <sup>-</sup> , and SO <sub>4</sub> <sup>2-</sup> )	0.5-liter polyethylene bottle	Cool to 4°C	28 days
Metals (Ag, As, Cd, Cu, Hg, K, Pb, Ni, Se, Mn, B, V, Zn and Total phosphorus)	0.25-liter polyethylene bottles	HNO <sub>3</sub> to pH ≤ 2, Cool to 4°C	6 months; except Hg is 28 day hold time
Arsenic Species (As(III), As(V))	HCl cleaned 0.25-liter polyethylene bottles	6M HCl, Cool to 4°C	28 days
Total Ammonia	0.5-liter polyethylene bottle	H <sub>2</sub> SO <sub>4</sub> to pH ≤ 2; Cool to 4°C	28 days
Total cyanide	0.5-liter polyethylene bottle	NaOH to pH ≥ 12; Cool to 4°C	14 days

### B.3.4 Sample Handling

All sample containers will be pre-cleaned. Preservatives, if required, will be added to the containers prior to shipment of the sample containers from the laboratory (pre-preserved) or added to the samples(s) in the field as needed to meet sample preservation requirements.

All sample containers for submittal for laboratory analysis will be placed in a strong, rigid-walled shipping container such as a heavy plastic cooler. The following outlines the packaging procedures that will be followed.

1. When ice is used, secure the drain plug of the cooler with tape to prevent melting ice from leaking out of the cooler.
2. Line the cooler with bubble wrap, as needed, to prevent breakage during shipment.
3. Check screw caps for tightness and, if not full, mark the sample volume level of liquid samples on the outside of their sample bottles with indelible ink.
4. Affix sample labels onto the containers and write sample number on container with indelible ink.
5. Wrap all glass sample containers in bubble wrap to prevent breakage.

All samples will be placed in coolers with the appropriate chain-of-custody form. All forms will be enclosed in a large plastic bag and affixed to the underside of the cooler lid. Empty space in the cooler will be filled with bubble wrap to prevent movement and breakage during shipment. Ice used to cool samples will be placed on top and around the samples to chill them to the correct temperature. Both samples and ice will be double-bagged in large plastic bags. Each ice chest will be securely taped shut with strapping tape; and custody seals will be affixed to the front and back of each cooler.

#### **B.4 PERSONNEL TRAINING**

All personnel directly involved with the Treatability Study will be provided with a copy of this Plan. Personnel will be trained in the requirements specified herein and provided ample time to read and become familiar with these requirements prior to beginning the testing program.

TABLE B-2  
LIQUID SAMPLE LABORATORY ANALYSIS REQUIREMENTS  
GROUNDWATER TREATABILITY STUDY WORK PLAN FOR THE FMC OU  
Page 1 of 2

Parameter <u>WQP</u>	Analytical Method Number	Method Type	Reporting Limit (mg/l)	Estimated Accuracy*	Precision **	Average Concentration of Constituent in Groundwater (wells 110, 146, and TW-9S)	Groundwater Cleanup Standards (mg/l)***	Pocatello POTW Pretreatment Limits
Fluoride	9056 (b) or 340.2 (c)	Ion Chromatography or Potentiometric, Ion Selective Electrode	0.1	75% - 125%	± 30%	0.30	4	32
Nitrate	9056 (b) or 353.2 (d)	Ion Chromatography or Colorimetric	0.1	75% - 125%	± 35%	6.63	10	NA
Total Phosphorus	6010B (a) or 365.2 (c)	Inductively Coupled Plasma / Mass Spectrometry or Colorimetric (ascorbic acid)	0.02	75% - 125%	± 30%	2.54	NA	7.0
Sulfate	9056 (b) or 375.4 (d)	Ion Chromatography or Turbidimetric	1	75% - 125%	± 30%	168	250	NA
Potassium	6010B (a)	Inductively Coupled Plasma Atomic Emission Spectrometry	0.1	75% - 125%	± 30%	43.4	NA	NA
Chloride	9056 (b) or 325.3 (c)	Ion Chromatography or Titrimetric	1	75% - 125%	± 30%	136.3	250	NA
Total Ammonia (NH3 + NH4 as N)	350.3 (d)	Potentiometric, Ion Selective Electrode	0.2	75% - 125%	± 30%	0.17	NA	NA
Parameter <u>Metals (mg/l)</u>	Analytical Method Number	Method Type	Reporting Limit (mg/l)	Estimated Accuracy*	Precision **	Average Concentration of Constituent in Groundwater (wells 110, 146, and TW-9S)	Groundwater Cleanup Standards (mg/l)***	Pocatello POTW Pretreatment Limits
Arsenic	6010B (a)	Inductively Coupled Plasma Atomic Emission Spectrometry	0.002	75% - 125%	± 30%	0.03	0.01	0.06
Arsenic(V)	1632 (e)	Atomic Absorption Spectrometry	0.001	75% - 125%	± 30%	Unknown	NA	NA
Arsenic(III)	1632 (e)	Atomic Absorption Spectrometry	0.001	75% - 125%	± 30%	Unknown	NA	NA
Cadmium	6010B (a)	Inductively Coupled Plasma Atomic Emission Spectrometry	0.002	75% - 125%	± 30%	<0.0005	0.01	0.2
Copper	6010B (a)	Inductively Coupled Plasma / Mass Spectrometry	0.01	75% - 125%	± 20%	0	1	0.5
Cyanide	335.4 (d)	Colorimetric	0.01	75% - 125%	± 30%	0.01	0.2	0.2
Lead	6010B (a)	Inductively Coupled Plasma / Mass Spectrometry	0.01	75% - 125%	± 20%	0	TT, 0.015	0.3
Mercury	SW 7470A (b)	Cold Vapor Atomic Absorbtion Spectrometry	0.0005	75% - 125%	± 20%	<0.0002	0.002	0.0006
Nickel	6010B (a)	Inductively Coupled Plasma / Mass Spectrometry	0.01	75% - 125%	± 20%	<0.04	0.73	1
Selenium	6010B (a)	Inductively Coupled Plasma Atomic Emission Spectrometry	0.0005	75% - 125%	± 30%	0.012	0.050	NA
Silver	6010B (a)	Inductively Coupled Plasma / Mass Spectrometry	0.01	75% - 125%	± 20%	<0.005	0.1	0.6
Zinc	6010B (a)	Inductively Coupled Plasma / Mass Spectrometry	0.02	75% - 125%	± 20%	0.001	71	1.2

**TABLE B-2**  
LIQUID SAMPLE LABORATORY ANALYSIS REQUIREMENTS  
GROUNDWATER TREATABILITY STUDY WORK PLAN FOR THE FMC OU  
Page 2 of 2

(a)	Analysis may also be performed using method 6020, both 6010 and 6020 from Test Methods for Evaluating Solid Waste, EPA SW–846, Third Edition, Update IIIB, as revised through 2002.
(b)	Test Methods for Evaluating Solid Waste, EPA SW–846, Third Edition, Update IIIB, as revised through 2002.
(c)	Methods for Chemical Analysis of Water and Wastes, EPA600/4–79–020, Revision, March 1983.
(d)	Methods for the Determination of Inorganic Substances in Environmental Samples (EPA/600/R-93/100).
(e)	Chemical Speciation of Arsenic in Water and Tissue by Hydride Generation Quartz Furnace Atomic Absorption Spectrometry (EPA Method 1632).
*	percent recovery
**	relative percent difference
***	Groundwater Cleanup Standards are Maximum Contaminant Levels or Secondary Standards per National Secondary Drinking Water Regulations. TT means Treatment Technique action level per the National Primary Drinking Water Regulations.
NA	Not Applicable; no POTW standard

**TABLE B-3**  
**SOLID SAMPLE LABORATORY ANALYSIS REQUIREMENTS**  
**GROUNDWATER TREATABILITY STUDY WORK PLAN FOR THE FMC OU**

<b>Parameter</b>	<b>Analytical Method Number</b>	<b>Method Type</b>	<b>Reporting Limit (mg/l)</b>	<b>Estimated Accuracy*</b>	<b>Precision**</b>	<b>TCLP Threshold (mg/l)</b>
Arsenic	1311	TCLP	0.05	75% - 125%	± 30%	5.0
Barium	1311	TCLP	0.05	75% - 125%	± 30%	100.0
Cadmium	1311	TCLP	0.05	75% - 125%	± 30%	1.0
Chromium	1311	TCLP	0.05	75% - 125%	± 30%	5.0
Lead	1311	TCLP	0.05	75% - 125%	± 20%	5.0
Mercury	1311	TCLP	0.01	75% - 125%	± 20%	0.2
Selenium	1311	TCLP	0.05	75% - 125%	± 30%	1.0
Silver	1311	TCLP	0.05	75% - 125%	± 20%	5.0

\* percent recovery

\*\* relative percent difference



**FMC Corporation**  
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Transmitted Via Email

May 23, 2014

Kevin Rochlin  
Project Coordinator  
U. S. Environmental Protection Agency  
1200 Sixth Avenue, Suite 900  
Seattle, WA 98101

Re: FMC Corporation Pocatello, ID  
Unilateral Administrative Order for Remedial Design and Remedial Action  
EPA Docket No. CERCLA 10-2013-0116  
Bench Scale Treatability Study Work Plan

Dear Mr. Rochlin:

As you are aware, FMC implemented the field portion of the Extraction Zone Hydrogeologic Work Plan (EZHWP) earlier this year. Groundwater samples were collected from each extraction well, as well as a composite sample collected during the 72 hour pump test, and sent for laboratory analyses pursuant to the EZHWP. The data collected during the implementation of the EZHWP will be used to proceed with developing the 30% remedial design for the groundwater remedy for your review later this year. Additionally, FMC has determined that the design will be furthered with some additional data to be developed in some bench scale treatability (jar) tests which will be conducted at two off-site laboratories.

A copy of the bench scale treatability study work plan is provided for your information. As we discussed, this treatability work is not being performed on-site at the FMC Plant OU and any agency comments on the treatability work will be provided with comments on the 30% remedial design report and addressed as the design proceeds.

Sincerely,

A handwritten signature in black ink, appearing to read "Barbara Ritchie", written in a cursive style.

Barbara Ritchie  
Associate Director, EHS

cc (as required under the UAO):

Doug Tanner, Idaho Department of Environmental Quality  
Scott Miller, Idaho Department of Environmental Quality  
Kelly Wright, Shoshone-Bannock Tribes  
Susan Hanson, Shoshone-Bannock Tribes